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Micrographic Observations of the Carbonaceous Mesophase by a Quenching Hot Stage

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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A hot-stage microscope with quenching capability was employed to relate the observations made at the free surface of a pitch during pyrolysis to the bulk microstructure. Freshly formed mesophase spherules display an "iceberg effect" with only a limited upper segment exposed to view. The mesophase layers tend to lie normal to the free surface, and $\pm 2\pi$ as well as $\pm \pi$ disclinations display discontinuous cores at the free surface; aside from these minor effects, surface observations appear to be representative of events taking place within the bulk mesophase.

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MICROGRAPHIC OBSERVATIONS ON THE CARBONACEOUS MESOPHASE BY QUENCHING HOT STAGE

At the Fourteenth Conference on Carbon, Hoover et al.[1] demonstrated by means of a film that dynamic observations of mesophase behavior were possible with the use of a hot-stage microscope with polarized-light reflected from the free surface of the pyrolyzing liquid. Although these observations confirm and extend the understanding of mesophase behavior as inferred from studies of polished sections of quenched specimens, there remains the question of just how closely the free-surface observations relate to the three-dimensional morphology within the bulk mesophase. For this reason we designed and constructed a hot-stage microscope with a quenching capability,[2,3] so that a specimen could be pyrolyzed to a point of interest as observed on the free surface, and then quenched to a solidified body that could be sectioned for detailed micrographic study.

Polarized-light micrographs of the free surface and a vertical section through the free surface are compared in Fig. 1 for a specimen of petroleum pitch (Ashland A240) pyrolyzed to approximately 445°C. The spherules at the free surface display an iceberg effect with only a limited upper segment exposed to view at the surface. Thus, most coalescence events begin below the surface and only appear when the coalesced region has reached the surface.

The micrographs also illustrate two further points relating observations on the free surface to those made on polished sections. The polarized-light response on the free surface provides sharper contrast and crisper definition of the extinction contours. Furthermore, the extinction crosses, which

[1]D. S. Hoover, A. Davis, A. J. Perrotta, and W. Spackman, Extended Abstracts, 14th Conf. on Carbon, 393 (1979).
[2]M. Buechler, C. B. Ng, V. L. Weinberg, and J. L. White, Carbon '80, Arbeitskreis Kohlenstoff der Deutsche Keram. Ges., 346 (1980).
[3]M. Buechler, C. B. Ng, and J. L. White, Extended Abstracts, 15th Conf. on Carbon, 182 (1981).

locate $\pm 2\pi$ disclinations,[4] usually appear with sharply pinched centers on the free surface and with the broad diffuse centers on polished sections.

The vertical section (Fig. 1) was mapped by polarized-light response[5] to obtain a structural sketch (Fig. 2). The large spherules display the coarse lamelliform morphology typical of fresh undeformed mesophase, but the layers at the free surface tend to lie normal to this surface. Examination of specimens quenched from various levels of pyrolysis confirmed this tendency but also indicates that this orientation of surface layers is readily disturbed by deformation of the mesophase, e.g., by bubble percolation. The layer curvatures necessary to orient the layers near normal to the free surface usually occur within a few microns of the surface. Thus, the strong contrast of polarized-light observations on the free surface of freshly formed mesophase results from the natural molecular orientation at this surface.

In conventional nematic liquid crystals, the rod-like molecular units tend to lie parallel to an interface (e.g., the interface with a glass cover slide), and the pinched crosses of the polarized-light extinction contours observed on this interface locate $\pm 2\pi$ wedge disclinations with discontinuous cores.[6] Meyer[7] has shown that within the bulk nematic liquid crystal, beyond the range of orienting forces near the interface, the molecules comprising the core of a $+2\pi$ wedge disclination tilt toward parallelism with the disclination line to form a core with continuous structure. In the carbonaceous mesophase, the tendency for the mesophase layers to stand normal to the free surface also requires the core of a $+2\pi$ wedge disclination to be discontinuous at the surface, but within bulk mesophase the core can relax to the cup-shaped continuous structure sketched in Fig. 3.

This situation is thus symmetrical with the saddle-shaped continuous structure of the core of -2π wedge disclinations (Fig. 4) deduced from

[4]J. E. Zimmer and J. L. White, Mol. Cryst. Liq. Cryst. 38, 177 (1977).
[5]J. Dubois, C. Agace, and J. L. White, Metallography 3, 337 (1970).
[6]S. Chandrasekhar, Liquid Crystals, Cambridge Univ. Press (1977).
[7]R. B. Meyer, Phil. Mag. 27, 405 (1973).

Fig. 1. Polarized-light micrographs of a quenched specimen of mesophase pitch as observed on free surface (upper view) and on a polished vertical section (lower view). Crossed polarizers.

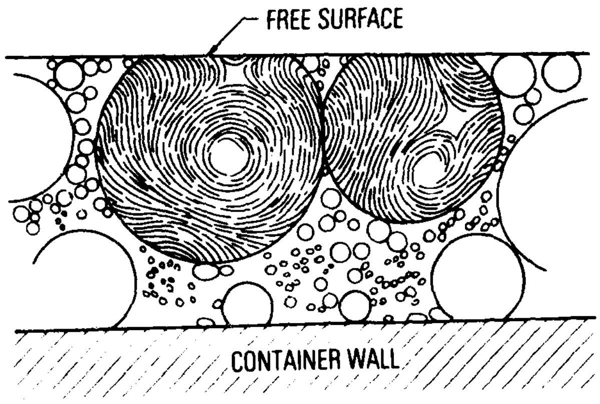

Fig. 2. Structural sketch of mesophase layer orientations in large spherules of lower view in Fig. 1

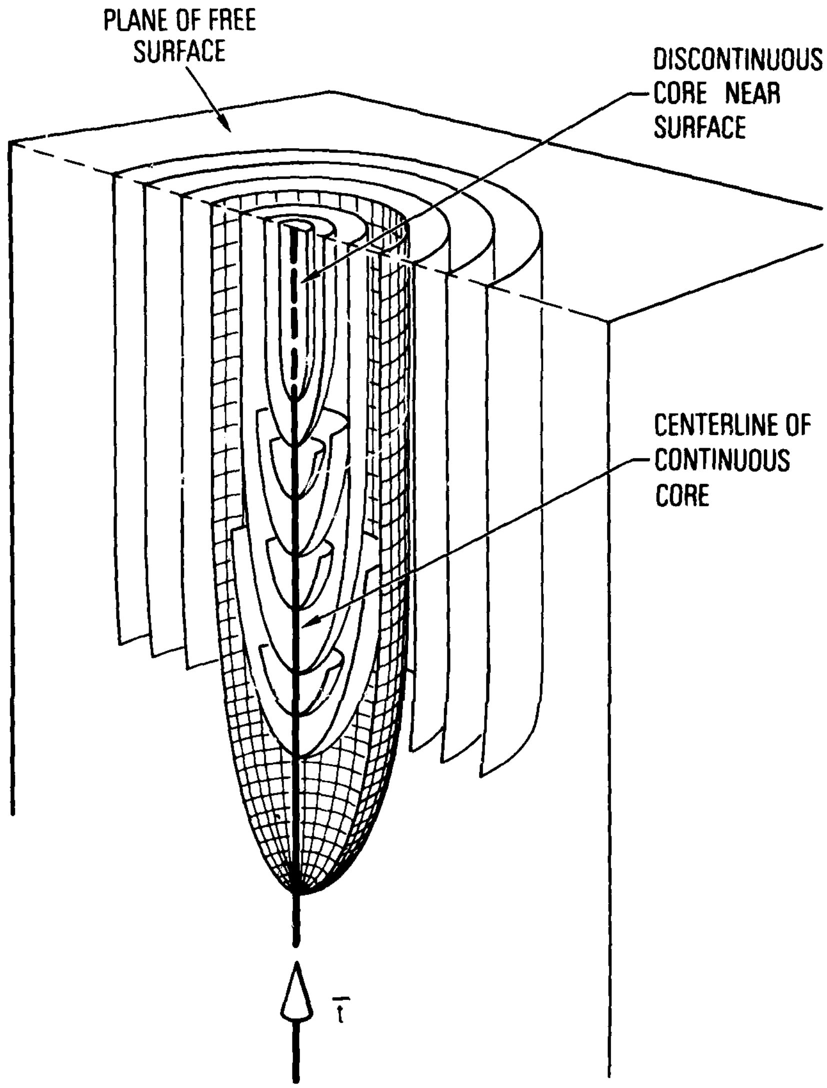

Fig. 3. Lamelliform model of a $+2\pi$ wedge disclination. A grid placed on a surface defined by the later orientations shows the cup-like structure of the continuous core. The core becomes discontinuous near the intersection with the free surface.

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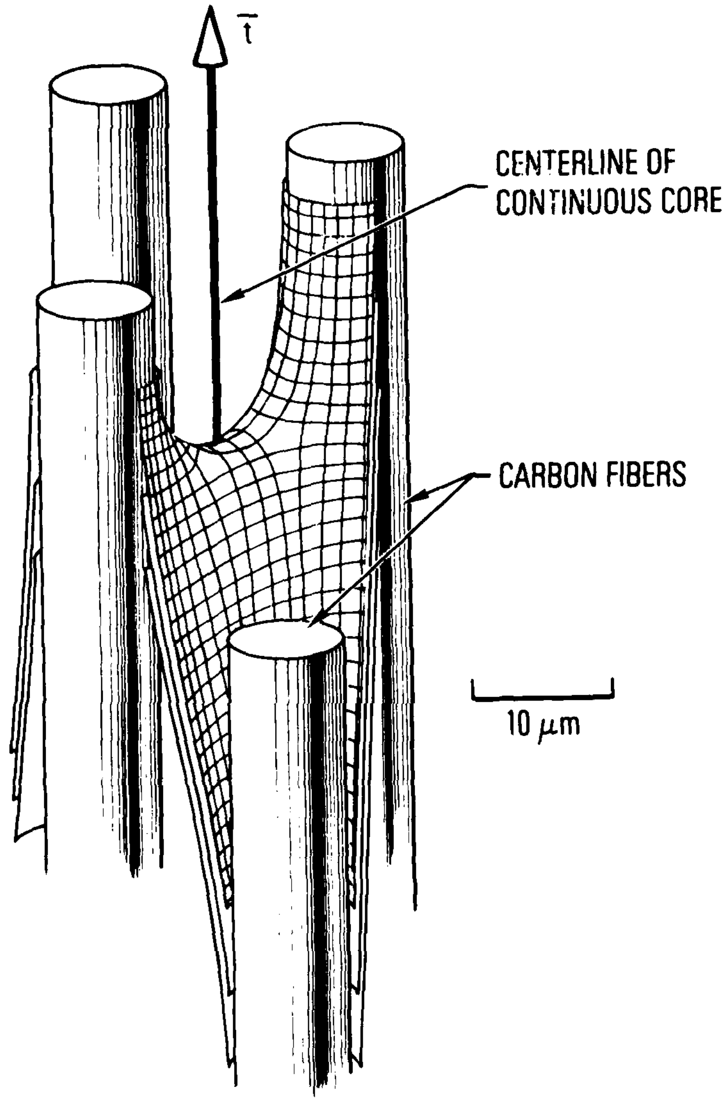

Fig. 4. Lamelliform model of a -2π wedge disclination formed in the mesophase matrix between four carbon filaments of a carbon-carbon composite.[8] A grid placed on a surface defined by the layer orientations shows the saddle-like structure of the continuous core.

micrographic study of certain fiber-reinforced composites.[8] Topologically, the carbonaceous mesophase seems to behave as a nematic liquid crystal, but with the vector defining the molecular orientation standing normal to the long dimension of the layer-shaped molecules rather than parallel as in the case of the rod-shaped molecules that comprise conventional nematic liquid crystals.

[8] J. E. Zimmer and J. L. White, Extended Abstracts, 14th Conf. on Carbon, 429 (1979).

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